Evaluating Packaging for Soil-Degradable Sensors

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***Abstract*— Internet of things (IoT) systems for precision agriculture have the potential to conserve water and increase agricultural productivity. Enabling these systems with a capacitive moisture sensor, which is fully biodegradable paper substrate with a cellulose nanofibrils surface, helps detect moisture levels of soil in agricultural fields. But these soil-degradable sensors degrade very rapidly without a packaging to protect the sensor. Therefore, a need to develop a biodegradable packaging that can effectively extend the lifespan of these sensors while maintaining sensor performance and functionality is required. Several packaging designs have been investigated to identify a design that will most effectively protect the sensor, and the mechanical properties of 3:1 beeswax: soy wax composite wax blend are analyzed. A packaging design and material are selected and recommended as an appropriate biodegradable packaging for these soil-degradable sensors.**

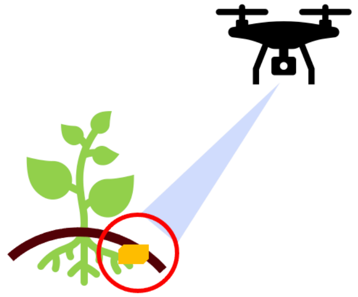
***Index Terms*— Internet of Things, adhesion, agriculture, beeswax, biodegradable, capacitive sensors, friction, grippers, manipulator dynamics, microsensors, material science, moisture, moisture measurement, radiofrequency identification, soil, soil sensors, soy wax, temperature sensors, wireless sensor networks**

I. INTRODUCTION

With agriculture as the largest pressure on the world's fresh water sources [1] and the global population projected to exceed 9 billion by 2050 [2], it becomes a pressing concern to efficiently conserve fresh water and sufficiently produce food. High spatial resolution monitoring of agricultural fields as used in Internet of Things (IoT) sensing systems can provide insight into soil conditions and offer deployment direction of resources. To achieve such an integrated network, inexpensive yet high accuracy sensors are required to equip such IoT systems for agriculture. The National Science Foundation Engineering Research Center for the Internet of Things for Precision Agriculture (Iot4Ag) launched its collaborative programs to develop this type of communication technologies and to improve current methods of real time monitoring in farms to tackle these food and water security needs.

High accuracy soil moisture and humidity measurements are critical for maximizing crop yield without wasting fresh water [3]. Studies show that state of the art soil-degradable sensors can effectively measure soil moisture [4]. These sensors respond to soil moisture, detecting changes in the levels of water in the soil. The capacitive sensors use silver ink on a cardstock paper substrate with a cellulose nanofibrils (CNF) surface in certain geometries [4]. When the cardstock paper substrate picks up moisture, the dielectric constant of the paper increases, respectively changing the capacitance measurement of the sensor.

These sensors are placed in the soil along crops, such as row crops like corn, and detect moisture levels in the soil. The sensors are directly used in the 902-928 MHz frequency range as chipless passive sensing systems embedded in the IoT network. The sensors are shown to have a self-resonance well above this operating frequency band.



However, these sensors are known to degrade very rapidly, and to extract useful information, these sensors need to last at least one crop season, around 6-9 months. This work focuses on understanding the degradation behavior of soil-degradable devices and developing a packaging that can prolong the life of the sensor and maintain the sensing functionality while simultaneously biodegrading within one crop season.

A packaging design for the sensor has not yet been studied. Because soil sensors need external environmental access to sense stimuli, the effect of semi-encapsulated packaging, with a window or an access hole, is required. However, the size of the window may affect with the device performance. Therefore, the effect of packaging design on overall device behavior and degradation has also not been explored and needs to be studied.

At the same time, a packaging material that is proven to be biodegradable within 6 to 9 months has also not yet been studied. Waxes are generally known to be biodegradable and can be considered as an effective material, but the specific properties of waxes also need to be studied [5, 6]. Beeswax with soy wax additives has been studied as cheese coating [7] and edible food packaging [8]. This research can be translated to measuring the mechanical properties to determine which material best fits our soil degradable sensor application.

A. Hypothesis

Unpackaged paper sensors are expected to rapidly degrade and fail when placed in the soil. The exact degradation timeline and rate are determined from collecting the electrical characteristics, specifically the phase and capacitance, of the sensors as a function of their time in the soil. With packaging, however, more fully packaged sensors with smaller window sizes are predicted to degrade much slower while retaining an accuracy of sensing. When exposed to moisture from the soil, there is a change in the real part of the phase, causing lower resistances among the no packaged and larger window sizes.

II. BACKGROUND

Soil-degradable sensors developed previously are constructed from CNF-coated paper and this paper can degrade, especially when exposed to moisture in the soil [4]. To expand the longevity of the soil sensors and protect the inked sensors, a biodegradable coating is required to enclose the sensor.

Specifically, a composite blend of beeswax and soy wax (BW: SW) has been shown as an effective encapsulant for soil-degradable electronic applications [9]. However, although specific BW: SW ratios have been known to further degradation rates in certain compost-soil conditions, rigorous mechanical characterization of the wax blend has not yet been studied.

To develop a biodegradable packaging that can sustainably prolongate the life of theses sensors, we need to first understand the behavior of the sensors without any packing in the soil. Then, the role of packaging on sensors needs to be studied and a type of biodegradable packaging material must be selected.

A. Current Studies

In current studies, the capacitance and phase of sensors buried in topsoil with 15% soil moisture to simulate a sandy loamy mixture is collected. These studies are occurred in this lab. However, the moisture in the soil creates a resistive interaction with the sensor. At low frequencies especially, the preferred behavior of current is to go through the soil, as a resistor, than through the capacitive soil sensor, producing capacitive rates that are ill-representative of the actual behavior of the sensor. A method that can truly capture the behavior of the soil sensors is required.

At the same time, it is important to understand how the sensor performs as it interacts with the environment. When exposed to moisture from the soil, a change in the real part of the phase and capacitance is seen. The changes in phase over time indicate the sensors performance as an ideal capacitor. Measuring capacitance allows characterization of sensor function over the degradation process. The greater the percent change in capacitance values of the sensors over time indicates how the device is degrading and failing as it no longer represents the original capacitance values. Both values are required to frame a full understanding of how packaging affects sensor degradation and performance.

Although packaging protects the device and enables functionality of the device for a longer lifetime, the varying effect of packaging from cardstock paper substrate alone to cardstock paper substrate with the CNF surface coating needs to be studies as well.

B. Packaging Material

As for packaging material, previous studies mention waxes as a viable inexpensive option for biodegradable packaging needs. Several materials have been previously studied as biodegradable packing, including paraffin wax, carnauba wax, soy wax, beeswax, gelatin, and cellulose. The advantage of using beeswax (BW) is that it supports farmers, is sustainably sourced, and is environmentally friendly while still biodegradable [5]. Simultaneously, soy wax (SW) is known for its hydrophobic properties and is also a great candidate for packaging applications because of its greater levels of stearic acid contribute to greater degradation of *n*-alkanes. On the other hand, paraffin wax is not biodegradable and carnauba wax is somewhat expensive.

Table 1 The common properties of these waxes is shown below.

Wax Types	Melting Temperature (ºC)	Temperature Range (ºC)	Viscosity (cP)	Ref.
Paraffin	46–68	60-100	2.3-1	[5]
Carnauba	83-91	135	6.7	[5]
Beeswax	63-65	60-100	11-3	[5]
Soy wax	49-82	45-53	-	[6]

As seen from Table 1, very minimal physical properties and mechanical behaviors of waxes is known. Narrowing down to BW and SW for the purpose of biodegradable packaging, more mechanical characteristics of these waxes, such as moduli and strain to failure, needs to be studied.

The effect of biodegradation of the composite blend of beeswax and soy wax (BW: SW) on their mechanical properties have not been studied previously in the literature. In this study, we aim to collect tensile, data of the BW: SW blend as it degrades in a highly organic compost soil environment over a 6-week period. We will then utilize this data to determine Young's Modulus and yield behavior to help predict how the wax will degrade over time.

C. *Importance of this Study*

Studying the material properties of this biodegradable coating for soil sensors is important as it builds the groundwork to help regulate the seasonal usage of sensors – approximately for 4 to 5 months. Not only does this project aid Iot4Ag goals but also will expand my understanding of applicable biomaterials and research methodology and discovery.

III. METHODOLOGY

To truly understand all the effects of packaging on the sensor performance, this study has been divided into an electrical testing and mechanical testing component. Within the electrical testing section, the effect of packaging has been studied on cardstock sensors and CNF coated sensors. Fig. 1 describes in detail the specific conditions, tools, and values being measured.

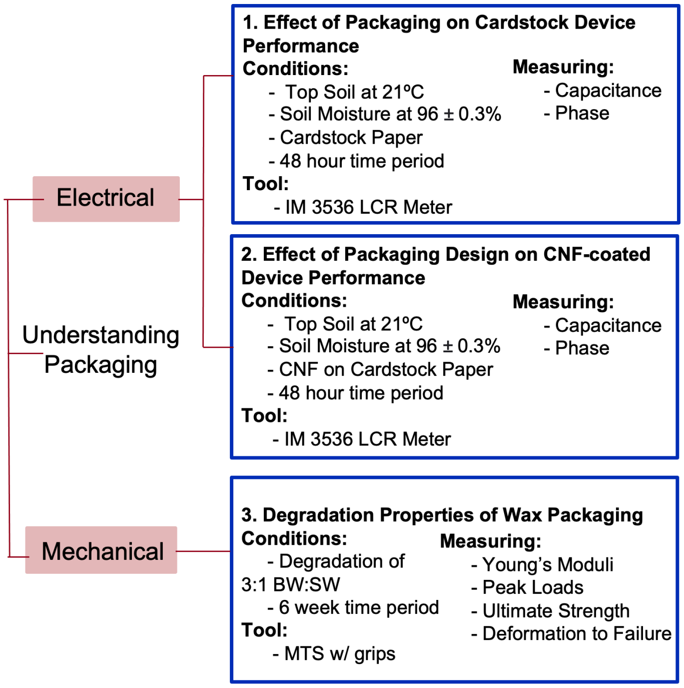


Fig. 1 Testing schematic.

A. *Electrical Testing*

1) *Effect of Packaging on Cardstock Device Performance*

As mentioned before, the moisture in the soil creates a resistive interaction with the sensor. At low frequencies especially, the preferred behavior of current is to go through the soil, as a resistor, than through the capacitive soil sensor, producing capacitive rates that are ill-representative of the actual capacitive values.

Therefore, two solutions are proposed to this problem. First, this issue can be resolved by collecting capacitor values at higher frequencies by using a Vector Network Analyzer. However, because this tool is currently unavailable for testing and usage, the testing procedure can be modified to remove the sensor from the soil to perform the electrical testing in air before returning it back to the soil at the end of data collection. This way, the sensor's performance can be compared to air as a baseline with consistent and reliable electrical data and it removes the resistive properties of the wet soil that were interfering with the data readings. As mentioned earlier, the capacitance and phase values will be measured.

Sensors will first be packaged with non-biodegradable Kapton tape with altering geometry of packaging types. Kapton tape is a thin polyimide film, which exhibits high thermal stability and electrical insulation properties. This allows us to understand how the sensor performs in a perfect sealed environment and remove any variables with how wax packaging might affect the sensor performance.

With the Kapton tape, there will be five different packaging types, each with 5 samples. This is 25 samples in total. The packaging types are no packaging, back packaging, small window size, medium window size, and large window size. With the window sized packaging, the entire device will be fully enclosed except for a window on the sensor so that it can interact with the environment. The small window size is 0.5 mm, the medium window size is 2.0 mm, and the large window size is 5.0 mm. The no packaged devices will represent the control group. Fig. 2 shows the methodology to create the sensors.

Fig. 2 In the first step, the picture the left, the sensors are printed. The second step, the middle picture, copper tape is attached. The next step, the right most picture, is to solder on wires. The last step, not pictured, is to add Kapton tape packaging.

First the paper sensors will be printed. Then copper tape will be used to make a waterproof connection between the sensor and external wiring. This wiring is required to place into the LCR benchtop meter to detect the capacitance and phase of the sensors. Back packaging devices have Kapton tape covering their entire backs. The windows are made from using biopsy punches to cut holes on the Kapton tape.

The sensors were pulled out of the soil to measure the capacitance and phase values with the benchtop meter. An IM 3536 LCR benchtop meter was used to measure the electrical data. The machine was calibrated before every use and the frequency was set at its highest, at 8 MHz.

The sensors were pulled out of the soil to measure the capacitance and phase values with the benchtop meter. This is because when the sensors are in sufficiently wet soil, the water in the soil starts to behave as a resistor. At low frequencies, the moisture in the soil behaves like a resistor in parallel, drawing current through this pathway and not through the capacitor. The readings from the water in the soil will dominate; therefore, it is imperative to remove the sensors from the soil and test the capacitance in air. Additionally, the sensors must be tested within 5 minutes of removal to ensure the moisture has not evaporated from the paper. This amount is within the recovery time constant [10]. As depicted by Fig. 3, two types of sensor geometry were used in creating the Kapton taped devices.

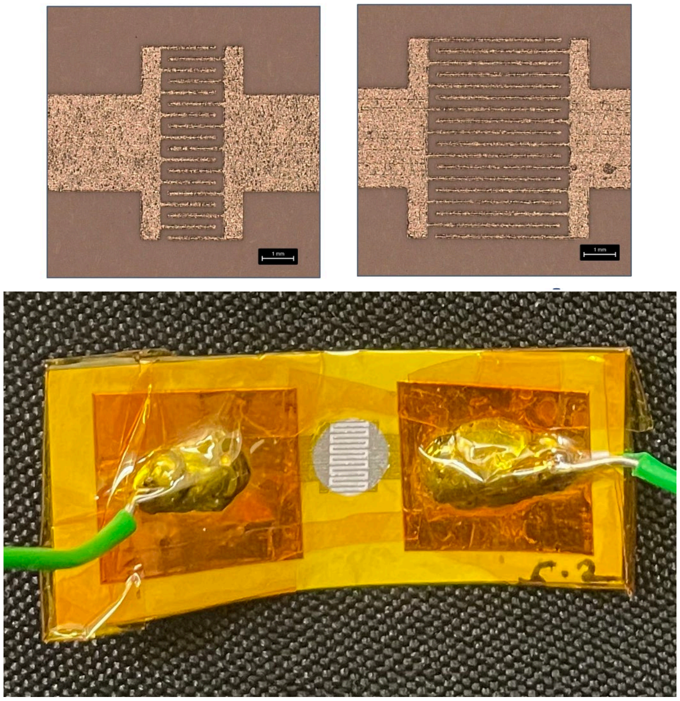


Fig. 3 The top two images show the geometry of the sensor. The capacitive values are 0.5 pF and 1.0 pF from left to right respectively. The bottom image is a fully assembled large window sized Kapton tape packaged sensor.

2) Effect of Packaging Design on CNF-coated Device Performance

The same methodology as this previous study was performed on CNF-coated sensors in the same exact manner. Again, we will collect capacitance and phase values of all three geometries of Kapton Tape packaged devices.

B. Mechanical Testing

3.) Degradation Properties of Wax Packaging

Because greater levels of stearic acid contribute to greater degradation of *n*-alkanes, it is imperative to continue to use a soy wax additive to beeswax. A composite 3:1 BW: SW was ultimately chosen because of the resulting Young's Modulus values when compared to other composite BW: SW blends.

The 3:1 BW: SW was hand poured into a PDMS mold of AHSS Type IV dog bone sample. The reference 3:1 BW: SW blend helps gain an understanding of a control group measurement without degradation yet. Six sets of ten 3:1 BW: SW samples have been buried in a high microbial compost soil bin. Compost soil is used to speed up the degradation process to see more degradation in six weeks than what degradation would occur in less microbial content soil like top soil. This bin is placed in a controlled environment with 15% humidity. A total of 70 wax samples were created.

This study will run for six weeks to assess how the sample degrades with all the same conditions. Every week, 10 new samples were recovered or unburied from the soil to perform tensile testing on. Using the tensile strength machine in the lab, the samples will be measured for elongation and load. These average values will be collected and compared with respect to the control and each week of degradation.

IV. RESULTS AND DISCUSSION

A. Electrical Tests

With the cardstock soil sensors, the experiment was held for 96 hours. At the start of the experiment, all samples represented a near ideal capacitor (Phase ~ -90º) when calibrated before burial into the soil, regardless of the packaging type. The starting capacitance of the samples varied significantly from the supposed capacitance value of the respective geometry. This is to be expected because the silver ink of the sensor is placed on a cardstock substrate and not a CNF coated cardstock substrate [10]. Moreover, to compare across the varying geometries and intended capacitances, the capacitance values were normalized and measured as a percent change in capacitance value to the original value collected at calibration.

Another starting condition important to point out is the relative humidity (RH %) of the soil, which was measured throughout the first 48 hours of the experiment to be 96 ± 0.3 %. By hour 96, the RH% of the soil decreased to 88%, which can be seen by the relative decline in both phase and capacitance of all the samples.

With longer periods of burial time, the more enclosed packaging types tend to no longer behave like an ideal capacitor, where the smaller sized window packaging and medium sized window packaging deviate the most from their original phase. This may be because the moisture from the soil that enters through the window remains trapped inside the packaging, further degrading the cardstock material and leading to device failure. While Fig. 4 shows the phenomenon that more enclosed devices fail, it also shows that large window sizes continue to maintain device performance by having least percent change in capacitance and closer to ideal capacitor phase values.

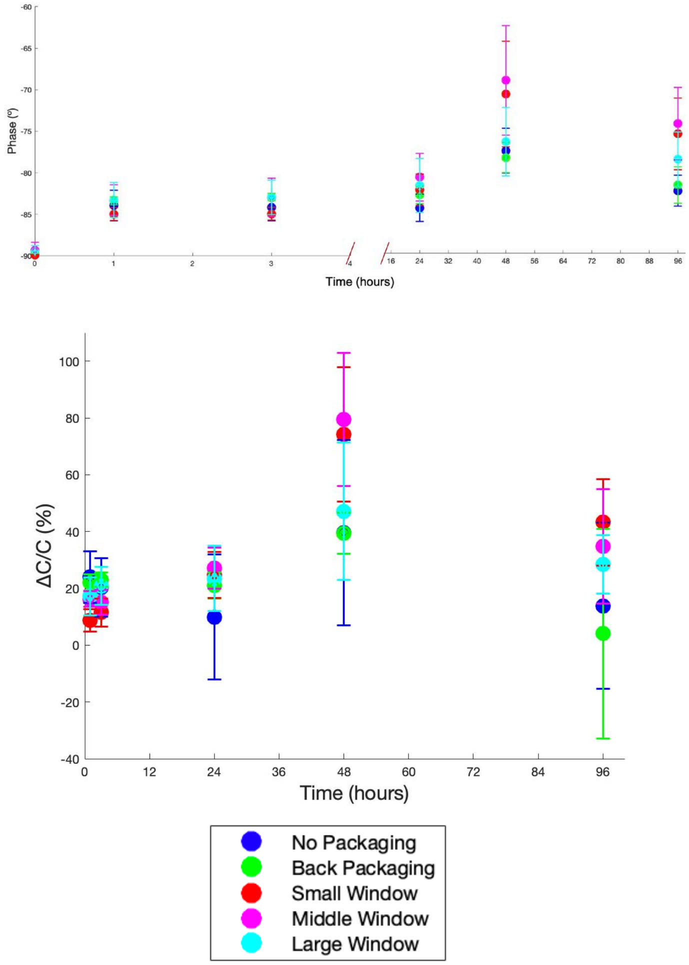
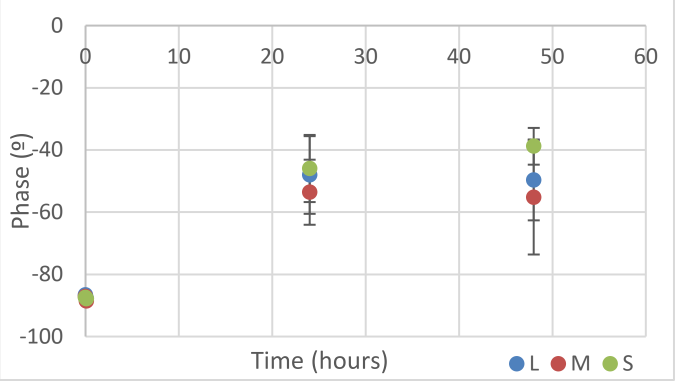


Fig. 4 The top graph show the phase values of the cardstock sensors. The lower graph shows the percent change in capacitance over time. A legend is shown at the bottom.

Similarly, the same trend can be seen in the CNF-coated cardstock sensors, which can be seen in Fig. 5. This can be explained by the same reasons as the cardstock sensor results.



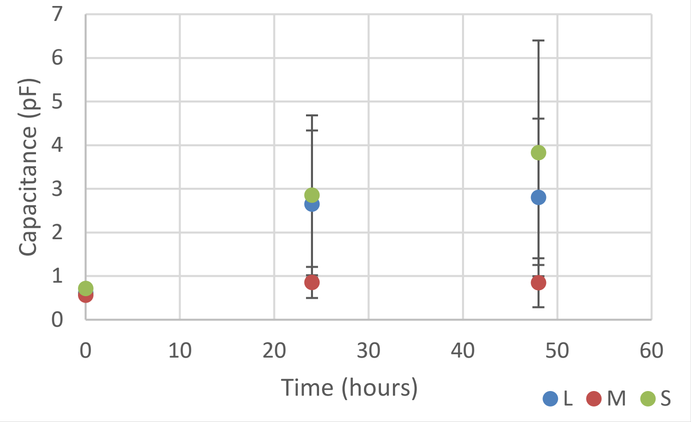


Fig. 5 The top graph show the phase values and the lower graph shows the capacitance over time of the CNF coated cardstock sensors.

Some limitations of this study that are important to note are the tools used; because the samples needed to be pulled out of the soil to collect capacitance values on the LCR benchtop meter, it becomes difficult to recover some of the response.

B. *Mechanical Tests*

Using the MTS tensile testing tool, the loading and extension values were measured from one set of reference samples and six sets of degradation samples. From these gathered values, Young's Modulus, average peak loads, and average extension was analyzed.

Fig. 6 shows the average moduli of about 10 samples from each week. Although the modulus seems to be inconsistent across the samples tested, there is a clear nonlinear growth. Interestingly, the elastic linear region that best categorizes the moduli of the wax blends is 0.2% as used in polymer study (see Fig. A1).

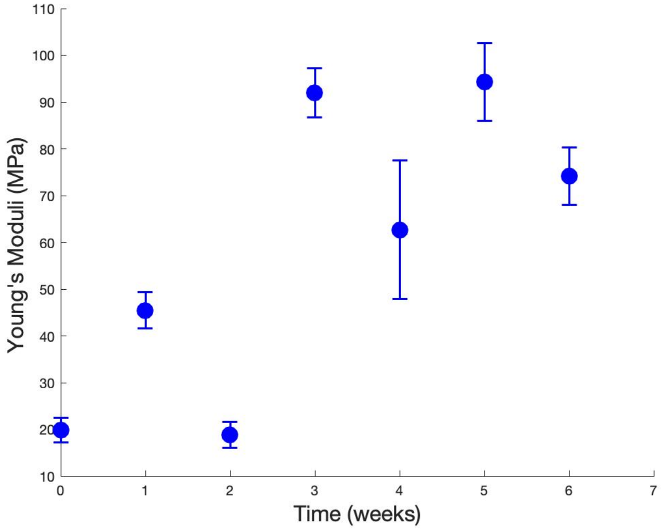


Fig. 6 Each data point for each week is an average of about 6-10 samples.

By week 3, the sample drastically changes in moduli, so to truly understand the degradation behavior, longer periods of degradation beyond six weeks need to be taken in the study.

To better understand the mechanical properties of degradation in waxes, the average peak loads and average extension were measured, which can be seen in Fig 7. These values were taken from about 10 samples within each week condition. It is important to note that because the individual cross-sectional area of each sample and the respective gauge length was not consistent during each experiment, the peak load and extension were analyzed instead of peak stress and strain to failure. The average extension represents deformation to failure.

According to the average peak load, the strength of the waxes remains relatively consistent when considering the errors. However, the deformation to failure decreases exponentially, meaning that the wax blend loses its ductility while gaining a brittle behavior.

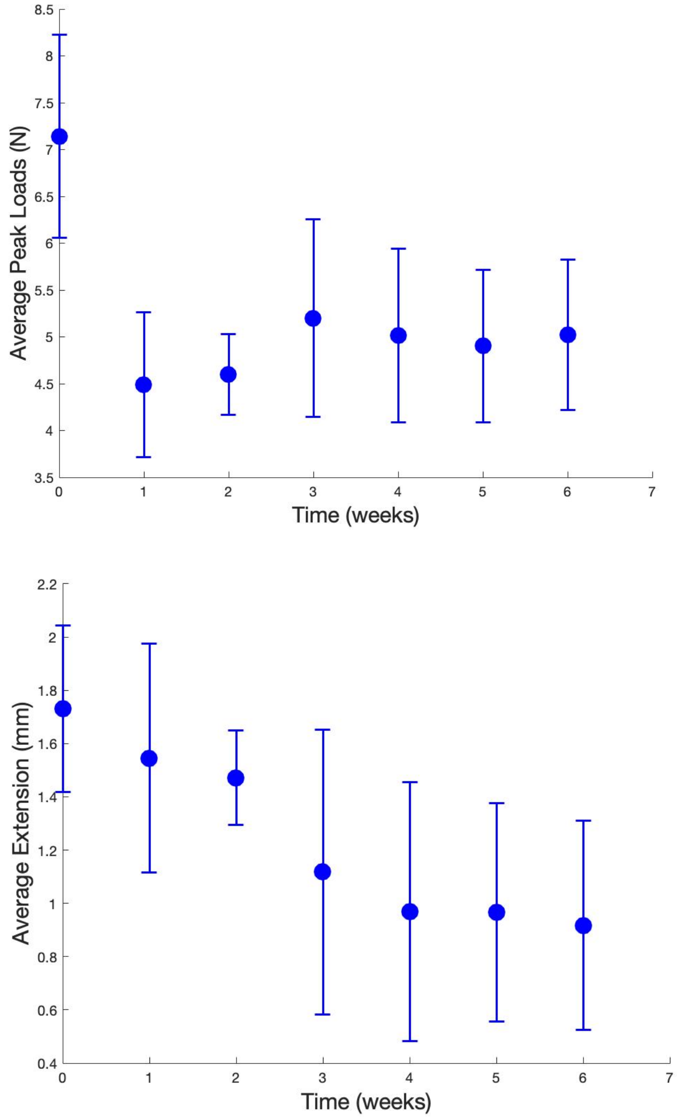


Fig. 7 Each data point for each week is an average of about 6-10 samples. The week 2 data point was rounded up to 2 weeks but actually only remained in the soil for about 1.5 weeks.

Qualitatively, the wax began to show signs of degradation as well. There is an observable change in color on the recovered samples where some spots on the samples with more opaque white in color than pale yellow. These spots of color variation seem to be random along the sample but may be due to different rates of degradation between the soy wax and the beeswax.

In addition to changing color throughout the degradation time, the wax samples from later weeks (3-6 weeks) were more often recovered already broken at the gauge region than the samples from the earlier week (1-2 weeks). These same later weeks samples were also easier to break at the gauge region after recovery from the soil due to mishandling and applying too much pressure during the cleaning process.

Lastly, the wax from later weeks seemed to have more soil stuck to the outer surface than the earlier weeks samples, as can be seen from Fig. 8. While this may be a sign of degradation, the stuck soil is harder to remove from these later weeks samples because these samples are more prone to breaking. This posed a limitation in comparing the mass before and after recovery from soil burial because the samples had some extra mass due to the stuck soil.

Fig. 8 The first sample is no degradation or the control group. The middle sample is from week 3 and the bottom sample is from week 6.

Another limitation from the system is that these wax samples were hand poured into a PDMS mold, meaning that there is a lot of variation in thickness and surface area between the samples. The average weight of the samples was 1.25 grams. The dimensions of the average cross-sectional area for the samples were 3.18 mm by 3.175 mm.

V. CONCLUSION AND FUTURE DIRECTIONS

This work focused on understanding the effects of packaging and certain packaging designs on soil-degradable moisture sensors. The study was split into two aspects: electrical and mechanical. Within the electrical study, the effect of packaging design on both cardstock sensors and nanocellulose-coated cardstock sensors was analyzed.

The packaged design explored 5 different packaging types: no packaging, back packaging, small window size, medium window size, and a large window size. The sensors were placed in soil and the capacitance of the sensor was measured as a function of degradation and soil moisture response. The response of the sensors was demonstrated over a timescale of several hours to a several days. To truly translate this study to their functionality in an agricultural field, sensors would have to collect data over an entire growing season, which typically lasts 9 months. Previous studies have shown that a sensor with no packaging degrades quickly, but a small window sized package is too enclosed and leads to moisture trapping and faster device failure. To truly test if moisture trapping is occurring, future studies need to collect the mass of each individual samples before and after. Packaging these soil-degradable sensors in a larger window size deems them as more functional ideal capacitors.

Additionally, tin future studies, the soil moisture can be measured instead of relative humidity. This can be done by controlling the weight percentage of water to soil, and is recommended to be checked consistently throughout the degradation time period.

With the mechanical testing, the 3:1 BW:SW composite blend proved to show some degradation within the 6 week time period. Tensile testing was performed, and load vs extension data was acquired. The moduli alone did not show a full picture of the degradation process, so it was important to extract average peak load and average extension values too.

Although this composite wax blend packaging is projected to reach some degradation in a high microbial content soil in 6 weeks, in order to understand how this packaging will last in top soil similar to that found in an agricultural field, further testing for longer periods of time is required. Degradation periods of 12 weeks or longer are now recommended to be studied.

Lastly, while studying the mechanical and tensile properties of wax, this study shows that waxes can be treated as polymers.

A. Wax Lamination

Future work will have to be conducted to also evaluate how the degradation process on a wax packaged sensor will impact sensor performance. By understanding the effect of non-biodegradable packaging on the sensor, fully biodegradable packaging will then be explored by replacing the Kapton Tape packing. The 3:1 BW: SW composite wax blend packaging will be used with the same experimental setup as the Tape Packaging experiment. This future direction will entail creating a PDMS mold to create a wax mold with specific window sizes in which the sensor can be placed. The methodology to laminate the wax will need to be derived. It would also be useful to understand how metallic nanoparticles in the soil can affect the degradation of the wax over time.

B. Nanoindentation

Due to time constraints, nanoindentation procedures were unable to be performed, but future studies can include in-depth studies of the various spots where color changes are noticeable. This would help identify if the wax composite blend is separating into its individual waxes and changing the behavior of the overall sample.

C. Future Applications

The findings of this study can be broadly applied as packaging materials for other IoT4Ag efforts, such as biodegradable batteries and phosphate sensors. One interesting avenue relating to biodegradable packaging that can also be explored is biodegradable food packaging and medical equipment packaging as well.

Lastly, while performing future studies, it is extremely crucial to remember to label all samples and collect the induvial masses of all samples before and after they were exposed to degradation. The copper tape offers no additive resistive powers. Silver paint and epoxide do not enhance the connection compared to the soldered wiring. In addition, regarding the wax samples, the individual surface area and dimensions of each and all samples needs to be collected to provide insightful stress-strain information.

APPENDIX

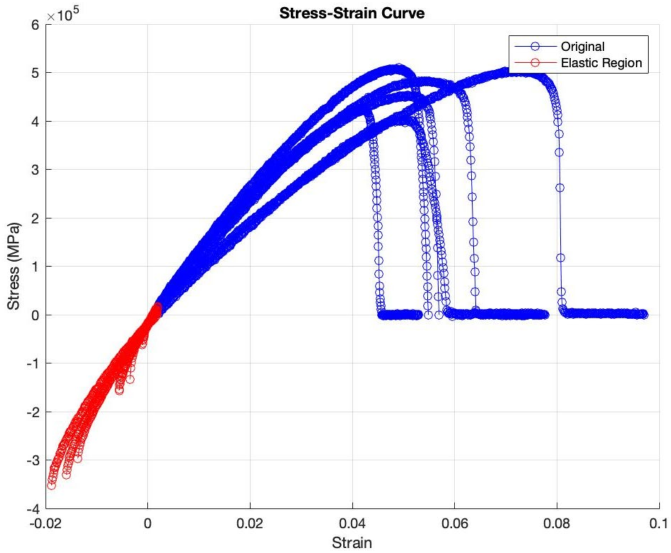


Fig. A1 Wax can be treated as a polymer with an elastic region of 0.2%.

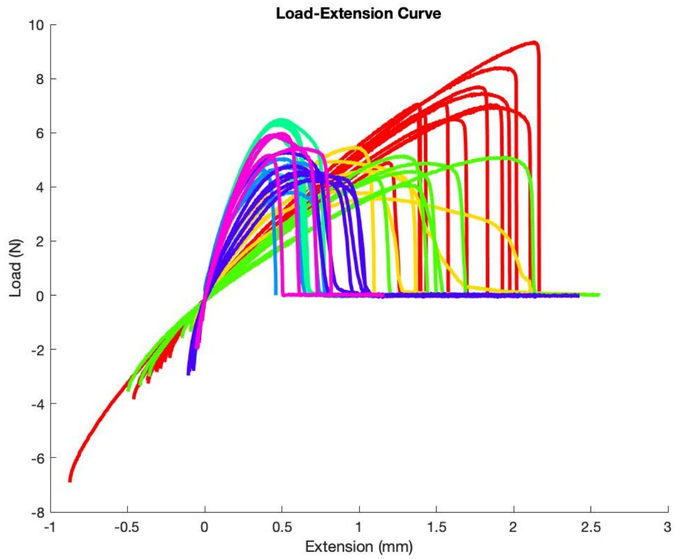


Fig. A2 Each color represents a new week, with about 6-10 samples representing each week. The order of the weeks starts from the control group of no degradation and is in rainbow order.

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